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DETERMINATION OF YTTRIUM IN HIGH DENSITY SILICON NITRIDE BY EMI--ETC(U)
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DETERMINATION OF YTTRIUM IN HIGH DENSITY SILICON NITRIDE BY EMISSION AND X-RAY FLUORESCENCE SPECTROSCOPY

BERNARD H. STRAUSS
POLYMER RESEARCH DIVISION

August 1981

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ABSTRACT

Two spectrographic methods were developed for the determination of yttrium in high density yttrium-doped silicon nitride. The first method employs emission spectrographic techniques which compares a solution of the dissolved sample with that of aqueous synthetic standards. The second method uses energy dispersive X-ray fluorescence techniques to compare fusion buttons made from the samples with those fabricated from pure yttria and undensified silicon nitride. Synthetic standards were required in both methods because there were no available yttrium-densified silicon nitride standards. Seven samples were analyzed whose yttrium content ranged from two to sixteen weight percent. Data on results are furnished.

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INTRODUCTION

High-strength silicon nitride, densified with yttria in weight percentages from 3 to 20% (2.4 to 15.8% yttrium), has been evaluated elsewhere as a structural material.^{1,2} Currently, it is being used to fabricate nozzles for 10-kW gas turbine power units and for various components of gas turbine engines.¹ It has high potential for applications in propulsion and power generation systems where high-temperature strength and resistance to oxidation, creep, and thermal shock are essential.²

Two spectrographic techniques were studied in this laboratory for the determination of yttrium. The first method is emission spectroscopy which compares a solution of the dissolved sample with that of aqueous synthetic standards.

The second method is energy dispersive X-ray fluorescence spectroscopy which compares fusion buttons made from the samples with those fabricated from pure yttria and undensified silicon nitride. Synthetic standards were required in both methods because there were no available yttrium-densified silicon nitride standards.

EXPERIMENTAL

Sample Preparation - Grinding Techniques

Samples were generally furnished to this laboratory in solid form, usually as rectangular rods with dimensions of 1/4" x 1/4" x 1", and had to be ground to small particle sizes as an initial step toward dissolution or fusion. A quick method of accomplishing this was to use a SIEBTECHNIK* high-speed grinder which enclosed the sample in a container lined with tungsten carbide and allowed a tungsten carbide "puck" to hit the sample at high velocity. An average sample was ground to 160 mesh size in less than twenty seconds. A disadvantage in using this grinder was the addition of tungsten to the samples. It appeared that the higher the yttrium content, the greater the tungsten addition. As much as 1% tungsten is added by grinding samples containing more than 7% yttrium.

Samples were also ground manually using a steel mortar and pestle. This eliminated any tungsten contamination but did add some adventitious iron which did not affect the yttrium determinations. Using this grinding apparatus, most samples had to be ground thirty minutes to an hour to achieve the desired particle size.

Emission Spectroscopy

Sample Dissolution and Preparation

Initially, 0.25-g portions of the 160-mesh powder samples were weighed directly into the removable Teflon liners of a Parr acid digestion bomb. Additions of 10 ml of hydrofluoric and 2 ml of nitric acids were made and the bomb sealed and placed in an air furnace at 150°C for at least 6 hours, usually overnight.

*Angstrom, Inc., 678 E. Huron River Drive, Belleville, MI.

1. *Ceramic Materials and Components for Small Automatic Gas Turbine Engine, Technology Assessment and Implementation Plan FY76-81*. Planning Directorate, Army Materials and Mechanics Research Center, April 1975.
2. GAZZA, G. E. *Effect of Yttria Additions on Hot-Pressed Si₃N₄*. Ceramic Bulletin, v. 54, no. 9, 1975, p. 778-781.

After cooling, the solutions were transferred to platinum crucibles. To remove silicon, five drops of sulfuric acid were added and the crucibles heated in a sand bath to copious fumes of sulfur trioxide followed by evaporation to dryness. After adding 10 ml of hydrofluoric acid and five drops of sulfuric acid, the contents were again evaporated down to fumes and dryness. This procedure was repeated twice more. The remaining residue was fused with 2 grams of sodium borate. After cooling, the sample was leached in 40 ml of 6N HCl. For samples containing less than 5% yttrium, fusing is not necessary although it is advised as mentioned in Results and Discussion. Such samples can be solubilized by the addition of 10 ml of 6N HCl and gentle heating. In order to use the same standards for fused and unfused samples, 2 grams of sodium borate dissolved in 30 ml of 6N HCl and 20 ml of water were added to the unfused samples. Both cobalt and gallium were found suitable as internal standards, although cobalt was used chiefly. In the case of cobalt, 8 ml of a 10 mg/ml solution was added to each unknown. For gallium, 1 ml of 20 mg/ml solution would be added. A final volume for all solutions of 100 ml was achieved by the addition of water.

Preparation of Standards

A series of synthetic standards were prepared containing yttrium in the range of 1 to 16% from a 10 mg/ml yttrium solution. This solution was prepared by dissolving 1.2699 grams of Y_2O_3 (Spex Industries) in 10 ml of 6N HCl and 5 ml of HNO_3 and diluting with water to a volume of 100 ml. Each standard solution contained the desired yttrium concentration, based on a 0.25-g silicon nitride sample and 2 g of sodium borate which were dissolved in 40 ml of 6N HCl and 20 ml of water. Cobalt was used as an internal standard.

A concentrated solution of 10 mg/ml of cobalt was prepared by dissolving 1 g of the metal in 20 ml of 6N HCl and diluting with water to a 100-ml volume. Eight ml of this solution was added to each standard solution, resulting in a cobalt concentration of 0.8 mg/ml at 100-ml final volume.

Method

Multiple spectra of the sample and standard solutions were recorded on photographic plates. Densities of the yttrium line 3195.6 Å and the cobalt line 3044.0 Å were measured by means of a microphotometer.³

A calibration curve of concentration versus change in logarithmic intensity ratio was prepared from the standard solution data. An example is shown in Figure 1. Such a curve was used to determine the amount of yttrium in the unknown samples. Listed in Appendix A are equipment, experimental, and photographic parameters for the emission spectrographic method.

X-Ray Fluorescence - Disk Preparation and Method

The powdered silicon nitride samples were fused in "non-wetting" platinum-gold crucibles (Alloy 7070, Engelhard Industries). Figure 2 contains a sketch of the crucibles and lid mold. To 0.50 gram of a powdered sample, in the special crucible, was added 5.0 grams of flux (lithium tetraborate) and 0.1 gram of internal standard (thorium nitrate). The crucibles were heated in an air furnace at 1050°C for six hours or overnight. Several times, while at temperature, the crucibles were swirled.

3. *Standard Practice for Photographic Photometry in Spectrochemical Analysis, Analytical Methods.* American Society for Testing and Materials, ASTM E116, Part 42, Philadelphia, PA, 1980, p. 24-46.

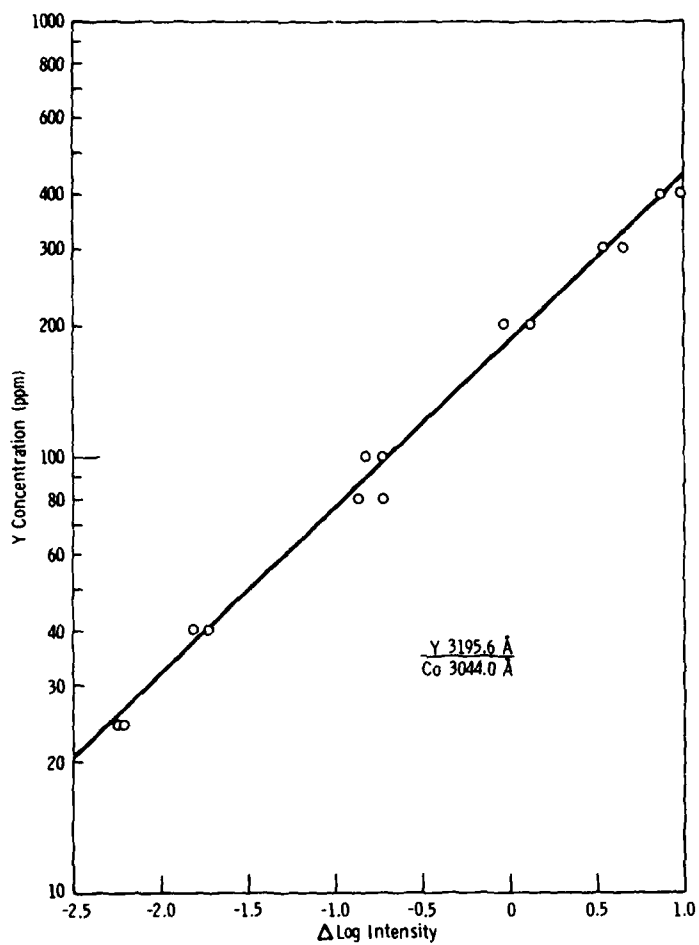


Figure 1. Typical analytical curve for yttrium using Co as an internal standard. Measurements by emission spectroscopy.

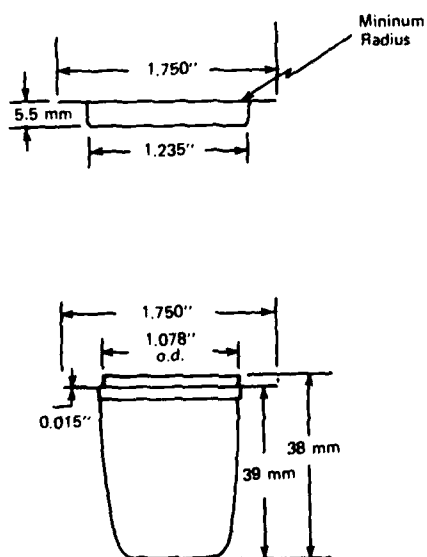


Figure 2. Platinum-gold crucible.

After the required heating, the lid-mold covers were removed (using long tongs) and placed next to its crucible. The contents were then poured into the lid-mold covers. The oven was shut off and the door left open until the temperature dropped to approximately 800°C. The door was then closed, allowing the residual heat of the furnace to increase to approximately 900°C. The lid molds were then furnace cooled to 200°C and then further cooled at room temperature to ambient. The disks could easily be removed from the lid molds by applying pressure to the center of the mold. This procedure produced melts that did not crack on cooling. Each disk measured approximately three centimeters in diameter by two millimeters in thickness.

Synthetic standards were prepared in a similar manner. "Pure" silicon nitride (containing no yttrium) was mixed with Y_2O_3 from Spex Industries to achieve the appropriate yttrium concentration. The same amounts of internal standard and flux, as used with the samples, were added.

The glassy disks (fusion buttons) were analyzed using an energy-dispersive X-ray fluorescence spectrometer. Intensities were obtained from integrated areas under the $ThL\alpha$ and the $YK\alpha$ peaks for each specimen. The yttrium counts were normalized by comparison to an average thorium count obtained for all disks analyzed. An intensity versus concentration curve is shown in Figure 3.

Appendix B contains the instrument parameters and the learn routine used for this work.

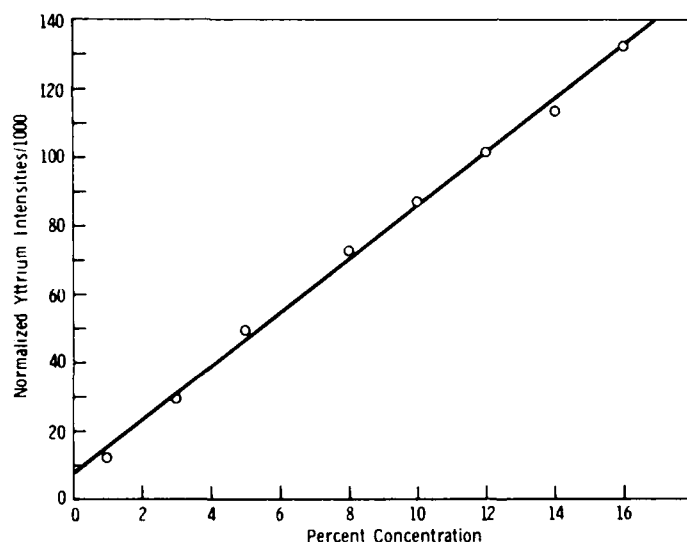


Figure 3. Synthetic standards in the form of glassy disks containing thorium nitrate internal standard. Intensities measured by X-ray fluorescence spectroscopy.

RESULTS AND CONCLUSIONS

This study lead to two instrumental methods devised for the determination of yttrium in yttria-densified silicon nitride. Both methods, emission spectrography and energy dispersive X-ray spectrometry, related the yttrium content of seven silicon nitride samples to those of synthetic standards, as no commercial standards were available. The seven samples ranged in yttrium content from two to sixteen weight percent.

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In the emission spectrographic technique, the dissolution of typical undensified silicon nitride was accomplished by treatment with a mixture of acids in a high-temperature, high-pressure bomb. However, for yttria-densified samples, this was not the case, as a gelatinous YF_3 precipitate was formed in the bomb. Such samples were fumed as described under Sample Dissolution and Preparation to remove the silicon, and various fluxes were tried to solubilize the remaining residue in order to obtain complete dissolution of the samples.

Initially, Na_2CO_3 was used as this had proved successful in solubilizing low concentrations of metallic impurities in silicon nitride.⁴ Later, a combination of Na_2CO_3 and $\text{Na}_2\text{B}_4\text{O}_7$ was tried. However, the optimum signal for yttrium was obtained using a straight $\text{Na}_2\text{B}_4\text{O}_7$ flux.

Attempts were made to solubilize the residue remaining after fuming, without resorting to fluxing. The residues were heated gently with 6N HCl. In most cases, the gelatinous precipitate remained and a clear solution could not be obtained. It was determined experimentally that complete solubilization would occur if the yttrium content was below 5%. Even then, the solutions appeared slightly cloudy, due partly to silicic acid and partly from complex tungstenic oxychloride compounds possibly present. Tungsten and chlorine were identified on a qualitative X-ray scan of the precipitate. The tungsten was introduced in the grinding of the specimens (Sample Preparation - Grinding Techniques). In most cases, however, the samples became clear after the addition of several milliliters of H_2O_2 and gentle heating. Slight cloudiness did not affect the yttrium determinations. Although use of a flux was found unnecessary for samples of less than 5% yttrium, it was decided that the same procedure should be used for all.

Both cobalt and gallium can be used as internal standards for yttrium, however, cobalt was used for the results reported in Table 1. Gallium is ideal if only yttrium is to be determined. A typical analytical curve using gallium is similar to that of cobalt. If other elements are also desired, then cobalt is a better choice as many more wavelengths of varying density are available. The disadvantage of cobalt is its presence in most silicon nitrides in concentrations of approximately 0.5%. The concentration of the internal standard was such that this amount became negligible.

Results obtained from the emission spectrographic method are shown in Table 1. Five determinations are reported for each sample, all obtained on separate days and

Table 1. RESULTS BY EMISSION SPECTROSCOPY

Sample	Results from Individual Disks	Average	1σ
30	16.5, 15.7, 15.8, 16.2, 16.2	16.1	0.33
43	14.3, 15.0, 14.4, 14.6, 14.8	14.6	.29
44	8.2, 8.4, 7.8, 8.5, 8.2	8.2	.27
45	8.4, 7.6, 7.8, 8.1, 8.4, 7.9	8.1	.31
46	4.2, 4.6, 4.3, 4.5, 4.0	4.3	.24
50	2.2, 2.2, 2.2, 1.9, 1.9	2.1	.16
68	11.6, 11.4, 11.1, 11.3, 11.2	11.3	.19

4. FERRARO, T. A., and STRAUSS, B. H. *Emission Spectrographic Determination of Metallic Impurities in Silicon Nitride by a Solution Method*. Army Materials and Mechanics Research Center, AMMRC PTR 73-5, April 1973.

from different powders of the same sample. Each day's results are the average of four individual sparkings of the same solution using a rotating disk electrode. Average values and one standard deviation from the average are shown.

Due to the geometric design of the energy dispersive X-ray fluorescence spectrometer, with the solid state detector placed directly under the sample, it was decided not to analyze solutions that may damage the detector. Therefore, glassy disks or fusion buttons were fabricated for use with this instrument. Duplicate disks were prepared from five of the samples and triplicate disks were prepared from the other two samples of higher yttrium content. Disks prepared from the same sample were analyzed to determine differences in their yttrium content. To do this, intensities were measured from the center (12-mm diameter being irradiated) of each of the disks. The highest difference in intensity between disks fabricated from the same sample was 5.5% relative, with the average difference being approximately 2.4%. The repeatability of measuring yttrium intensities from a single disk was determined by calculating the relative standard deviation obtained from five consecutive intensity measurements from a sample area (12-mm diameter) on a disk. The highest relative standard deviation obtained was 0.9%, with most values less than 0.5%.

The disks were next checked for segregation by measuring yttrium and thorium intensities in four different areas of the same sample, each 12 mm in diameter. Seven disks were randomly selected for these tests. It was found that the greatest percent difference in intensity between four areas on the same sample was 1.1%.

Results obtained in analyzing the disks are shown in Table 2. These results were acquired by measuring yttrium K α and thorium L α intensities for each unknown composition disk (Samples 30-68) and by use of the graph shown in Figure 2. The values for the parameters of this graph were obtained from synthetic standards previously described containing from one to sixteen percent yttrium. The yttrium values were normalized by comparison to an average thorium value obtained from the eight synthetic standards analyzed. The curve shown has a correlation coefficient of 0.998.

A comparison of results is shown in Table 3 between values obtained by emission spectrometry and X-ray fluorescence spectrometry. Agreement was fairly good for all seven specimens.

Both methods appear equal in reliability and the amount of over-all time required for analysis. However, the X-ray technique is preferable because less of the analyst's time is needed and the synthetic standards are reusable.

Table 2. RESULTS BY X-RAY FLUORESCENCE SPECTROSCOPY

Sample	Results from Individual Disks	Average
30	16.3, 15.5, 15.3	15.7
43	14.5, 14.7, 15.1	14.8
44	8.3, 8.2	8.2
45	8.1, 8.3	8.2
46	4.6, 4.3	4.4
50	2.2, 2.0	2.1
68	11.8, 11.4	11.6

Table 3. COMPARISON OF RESULTS

Sample	Emission Spectroscopy	X-Ray Fluorescence
30	16.1	15.7
43	14.6	14.8
44	8.2	8.2
45	8.1	8.2
46	4.3	4.4
50	2.1	2.1
68	11.3	11.6

RECOMMENDATION

It is recommended that both the emission and X-ray spectrochemical methods described in this report be included in Army purchase descriptions and future military specifications for yttria-densified silicon nitride.

APPENDIX A. EMISSION SPECTROGRAPHIC EQUIPMENT AND OPERATING PARAMETERS

Equipment

Baird 3-meter spectrograph, 2.75 A/mm reciprocal linear dispersion.
Jarrell-Ash Varisource.
Jarrell-Ash Microphotometer.

Experimental Parameters

Spark - 18,000 volts A.C., 4 breaks per half-cycle, 3-1/2 amperes
Analytical gap - 3 mm
Auxiliary gap - 6 mm
Sample electrode rotating disk - Ultra Carbon 106
Counter electrode - Ultra Carbon 108
Spark duration - 30 seconds
Pre-spark duration - 20 seconds

Photographic Parameters

Plates - Eastman Kodak SA 1
Developer - Eastman Kodak D19
Developing time - 3.5 minutes
Developing temperature - 20°C

APPENDIX B. X-RAY FLUORESCENCE INSTRUMENT PARAMETERS AND LEARN ROUTINE

Equipment

Finnigan 900A Energy Dispersive X-ray Fluorescence Spectrometer

Instrumental Parameters

Power - 20 kV, 1.0 mA
Acquire Time - 100 seconds
Pulse Pile up Rejection - approximately 30%
Vertical Scale Potentiometer - 12 K
Gain - 4
Fine Gain - 3
Base Line - 0
Post Gain - 1

Learn Routine

The learn routine is executed by push-button operations that sets up a program in the dedicated computer. Start/span positions varied very slightly in a day-by-day basis for the elements scanned. This is true only if baseline and fine-gain potentiometers are set at constant value. The following LEARN ROUTINE was used:

Learn
First Half Clear
Acquire (Data Acquired For About 20 Seconds)
Acquire (Stops Acquiring)
Start-Span Select
Start/Span

Measured Energy Ranges (keV)

Element	Peak	Peak Scan	BKG	BKG
Th	13.19(L)	12.95-13.42	12.14-12.48	14.02-14.36
Y	15.16(K)	14.89-15.43	14.02-14.36	18.18-18.52

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